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THERMAL PLASMA CHEMICAL SYNTHESIS OF POWDERS

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Thermal plasma processing has been increasingly used to synthesize submicron powders of high-purity ceramics and metals. The high temperatures generated with the plasma provide a vapor phase reaction zone for elements with high boiling points and refractory materials. An overview is presented on the general plasma technology used in synthesis and on the properties of plasma powders.

INTRODUCTION

The preparation of ultrafine ceramic and metallic powders is a growing area of research for the development of high-technology ceramics, metals, and cermets. Submicron powders are desired for the fabrication of materials with a lower sintering temperature (hopefully, without sintering aids) and a finer microstructure for reproducible and superior chemical and mechanical properties. Thermal plasma chemical synthesis is a viable method for producing the ultrafine, ultrapure powders needed for advanced materials research.

Plasma chemical synthesis of powders is an expanding area of research in the broader field of thermal plasma processing of materials. This technique offers the advartage of a single-step, continuous process, utilizing a large variety of reactants. Ceramic and metallic powders are formed in a vapor phase reaction within a high temperature gas (<10,000 K) generated by the thermal plasma. Plasma synthesis can produce ultrafine (<100 nm) and ultrapure powders, often in a metastable high-temperature phase, by rapid quenching of the hot synthesis gases.

The first section of this paper will briefly describe plasma reactor technology commonly used by current researchers. We will then review the current progress in the plasma chemical synthesis of ceramic (oxide and non-oxide) powders, metals, intermetallic alloys, and metal/ceramic composites (cermets). The review will cite a limited number of selected references as examples of plasma synthesis by injecting fine particle or gaseous reactants directly into the plasma or its tail flame. Broader reviews of plasma chemical synthesis can be found in the recent papers of Young and Pfender [1] and Akashi [2].

THERMAL PLASMA REACTORS

A plasma synthesis process generally includes five major parts: plasma generation, reactant species selection and injection, reactions within the plasma gas, quenching of the reaction products, and collection of the reaction products. Any process design will basically incorporate all of the above components, but the overall design will typically be governed by the type of plasma generation chosen.

Two basic methods of plasma generation are utilized in powder synthesis. One popular method is the dc arc plasma [1,3] formed by a high current flow between two electrodes; however, numerous variations of the arc plasma have been reported [3]. The other method is the rf inductively coupled plasma [4] generated by an electrodeless high-frequency excitation of the plasma gas. The rf reactor in use at Los Alamos [5] with a novel plasma tube is shown in Fig. 1. The main advantages of the rf reactor are the absence of electrode corrosion by the reactants and contamination in the product and the ability to inject reactants axially. In contrast, the dc plasma jet has a larger thermal efficiency with less capital cost, higher plasma temperatures, and greater stability during cold gas and particle injection. Some researchers have successfully combined the dc plasma jet and the rf plasma to form a hybrid plasma reactor [6] with the advantages of both plasmas. The thermal plasma generated by either method is generally operated at pressures near an atmosphere with high power levels (1 to 10^3 kW). Under thermal conditions the electron temperature and the heavy particle temperature are nearly equal [3,4], yielding temperatures of many thousand Kelvin and the high enthalpy gas needed for powder synthesis. The thermal and transport properties can be controlled by the type of plasma gas used (e.g., H_2, N_2, or Ar).

In a reactor using either plasma type, reactants can be fed into the excitation region with the plasma gas or into the jet or tail flame extending downstream of the plasma. In the case of an rf plasma reactor, reactants may be injected directly into the tail flame by a water-cooled axial probe [5] passing through the center of the plasma (see Fig. 1). Choice of injection method will be dependent on the chemical compatibility of the reactants with the electrodes and on the reaction chemistry. For example, silicon nitride [5-7] is commonly formed with greater yields by injecting ammonia after the plasma rather than by premixing ammonia or nitrogen with the plasma gas. Also, solid reactant can be introduced into the plasma gas before the plasma to provide a longer residence time and greater heat transfer to vaporize the reactant particles. Reactant powders are usually delivered to the reactor by elutriation in a fluidized bed, screw feeder, metered-cup wheel feeder, and other feeders commonly used in plasma flame spraying; however, little information on powder feeders is available in the synthesis literature [1].

A large variety of simple reactants can be introduced into the plasma, ranging from elemental and binary solids to gaseous hydrides,

halides, and diatomics. More complex organometallic, organic, and inorganic reactants are also possible candidates. However, the choice of reactants will largely be based on economics, reaction chemistry, or reactor compatibility.

Quenching plays a major role in plasma synthesis by terminating the reaction, capturing a high temperature phase as a metastable room temperature material and partially controlling the size distribution of the submicron powders. By proper design, quench rates can be varied from approximately 10^3 to as great as 10^8 K/s [1,2]. The main methods of plasma quenching [1] include mixing with a cold gas, injection into a fluidized bed, contact with a cold surface, and evaporation of a liquid spray.

The rapid reaction rates and short residence times for the reactants allow for the continuous injection of reactants into the plasma reactor and subsequently for the continuous collection of plasma powders. Particle collection devices [1] include bag filters, electrostatic precipitators, cyclone separators, and cold surfaces. The collection over the period of many hours of fluffy, ultrafine powders with a tap density less than 0.1 g/cm^3 can be challenging due to the great bulk of the powder and the need to collect without exposing the powder to air.

PLASMA CHEMICAL SYNTHESIS OF POWDERS

The development of plasma chemical synthesis for the preparation of ceramic and metallic powders probably represents the broadest and most intensive application of thermal plasma technology to chemical vapor deposition. This is an area which has identifiable needs for the specialized powder morphology produced in plasma vapor reactions and where the potential for significantly improved ceramics and metals (in terms of mechanical and chemical properties) outweighs the increased capital and operating costs associated with plasma synthesis. The following discussion addresses only the production of specialized, ultrafine powders which are sought for their increased surface energy over coarser powders to obtain sinterability at lower temperatures and a finer microstructure. However, the greater specific surface areas (>1 m^2/gm) for these powders also result in greater pyrophoricity and health hazards due to inhalation. At Los Alamos the handling of ultrafine powders is carried out by collecting powders in a vacuum-tight container for transfer to an inert atmosphere, when desired, and passivating through controlled oxidation before air exposure. Due to the potential toxicity of specific materials like nickel, full-face respirators are worn during reactor clean-up and collection of passivated powders.

NITRIDES. The synthesis of a wide variety of nitrides has been carried out both in rf induction and arc plasmas. These preparations are summarized in Table I. The most important feature of nitride

Table I. Plasma Production of Ultrafine Nitrides

Compound	Starting Materials	Plasma Type	References
Si_3N_4	$SiCl_4+NH_3+H_2$	RF/Arc	6,7,8
Si_3N_4	SiH_4+NH_3	RF	5,9
Si_3N_4	$Si+N_2/NH_3$	RF/Arc	7,10,11
AlN	$Al+NH_3$	RF	10,16,17
TiN	$TiCl_4+N_2+H_2$	RF	14,15,18
TiN	$Ti+N_2$	RF	19
ZrN	$Zr+N_2$	RF/Arc	12,14
TaN	$Ta+N_2$	Arc	12,13
MgN	$Mg+N_2$	Arc	12
NbN	$Nb+N_2$	Arc	13
VN	$V+N_2$	Arc	13
HfN	$HfCl_4+N_2+H_2$	RF	14
BN	$BCl_3+N_2+H_2$	RF	15,51

synthesis is the refractory properties of the final product. Those nitrides with high dissociation temperatures (Ti, Zr, Ta, etc.) can be successfully prepared from a nitrogen plasma while those with low dissociation temperatures (B, Si, Al) require the addition of large excesses of NH_3. Synthesis attempts under other conditions (e.g., [7] for Si_3N_4 and [15] for BN) yield powders which are either extremely substoichiometric with respect to nitrogen content or highly contaminated with the elemental material ($\geq$50%). It is pointed out by Kheidemane et al. [11] that in the case of Si_3N_4 the temperature range where nitrogen is chemically active as an atomic or molecular species is significantly above that for the decomposition of Si_3N_4. Thus, under normal conditions in the plasma, chemically active nitrogen and Si_3N_4 will not coexist. This was demonstrated by introducing a quench gas into the plasma at various heights in order to cool and recover products which might not otherwise be stable at that temperature. As the quench was moved up into the hotter regions of the tail flame where nitrogen is expected to be active, an excellent yield of Si_3N_4 was quenched out of a plasma containing Si and N_2, supporting the above hypothesis.

The bulk of the interest in the plasma synthesis of nitrides has focused on Si, Al, and Ti as a result of their mechanical and chemical properties. In general, Si_3N_4 has been found to be extremely fine (5-30 nm particle size) and crystallographically may be present in both α and β structures as well as in the amorphous form. The exception to this is the material produced by quenching from the $Si+N_2$ reaction [11] which is in the 50-800 nm size range. Production using $SiCl_4$ and NH_3 risks contamination with NH_4Cl [7]; however, this possibility can be reduced by employing a heated powder collector or

by replacing $SiCl_4$ with SiH_4 [5,9]. The ultrafine Si_3N_4 powder has been found to be extremely reactive in air, undergoing a substantial adsorption of oxygen and an associated loss of nitrogen [5]. The formation of TiN is not subject to NH_4Cl contamination since it is formed directly from nitrogen. Particle sizes from 5-150 nm have been observed with agglomerates as large as 10-30 μm. In order to drive the process to completion, very high N/Ti ratios are consistently required with some investigators going to N/Ti = 190. This might suggest that even at the higher temperature of formation for TiN (compared to Si_3N_4) only a small fraction of the nitrogen is in a chemically active form. In addition, when $TiCl_4$ is used as the titanium source, a substantial excess of hydrogen is required; however, the oxygen contamination present when Ti metal is used seems to be largely absent. AlN has been reported in the 50-70 nm size range [16] and when synthesized with an excess of NH_3, AlN fractions as high as 95% have been prepared [17].

CARBIDES. The synthesis of carbides is in some respects more straightforward than that of nitrides since carbides in general have much higher dissociation temperatures and carbon is available in an active form at lower temperatures than nitrogen. A sampling of the carbides described in the literature is shown in Table II. A second significant difference between nitride and carbide synthesis is disclosed on examining the range of starting materials utilized. In carbide synthesis, the possibility exists of utilizing carbothermic reduction; thus, it is possible to use an oxide as the starting material rather than the element or one of its halides. This has very important economic consequences since, in general, oxides are substantially lower in cost than the elemental or halide form. However, if an oxide is chosen, particular attention must be given to the purity of the product. In the case of SiC formation [21] by reduction with methane, the product is reported to contain large quantities of Si and SiO_2, possibly due to the short residence times inherent in most

Table II. Plasma Production of Ultrafine Carbides

Compound	Starting Materials	Plasma Type	References
SiC	CH_3SiCl_3	RF/Arc	20,23
SiC	SiO_x+CH_4	Arc	21,22
SiC	SiH_4+CH_4	RF	5,9,24
WC	$W+C/W+CH_4$	Arc	26,27
WC	W_3O+CH_4	Arc	25
TiC	$TiCl_4+CH_4+H_2$	Arc	28,29,32
TaC	$Ta+CH_4$	RF	31
TaC	$TaCl_5+CH_4+H_2$	Arc	30
B_4C	$BCl_3+CH_4+H_2$	RF	46

plasma powder operations. Kong et al. [22] report complete conversion of SiO when both methane and elemental carbon are used simultaneously; however, the resulting powder contains an excess of free carbon.

The most concentrated effort in carbide synthesis has focused on Si, W, and Ti for potential structural and tribological applications. The expectation is, as with some nitrides and oxides, that high-purity, ultrafine materials may be sinterable without additives. It is generally anticipated that this will lead to an improved microstructure and superior mechanical properties. Silicon carbide is usually formed in the β-phase with a particle size of 10-30 nm, although Kong et al. [22] report that their material prepared by carbothermic reduction is composed of particles up to 300 nm. Typical particle sizes and agglomerates in SiC prepared from silane and methane are shown in Fig. 2. As noted above, purity is highly dependent on starting materials. In the case of material prepared from silane and methane [9,24], total impurity levels (excluding oxygen) of less than 200 ppm have been achieved. The production of tungsten carbide has generally resulted in particle sizes from 1-30 nm; however, phase purity is a substantial problem. Most investigators report substantial amounts of one or more of the following phases along with β-WC: W, W_2C, C, or W_3O. Ultrafine carbides, although not pyrophoric in most cases, may nevertheless be extremely air sensitive. Studies on the air exposure of 10-30 nm SiC at Los Alamos [9,24] have shown oxygen adsorption of up to 5 wt% at ambient temperature. Also, this SiC powder can be readily oxidized to completion at temperatures below 1300 K.

OXIDES. Unlike ultrafine carbides and nitrides, oxides and oxide mixtures produced by plasma reactions tend not to be air sensitive. Exceptions to this can result from either water adsorption or the presence of unreacted starting material condensed on the surface. In general, this stability allows handling of these powders without the extensive and expensive precautions required for the carbides and nitrides. A summary of oxide and composite synthesis is presented in Table III. As can be seen from examining this table, the range of starting materials for oxide production is much broader than that for either nitrides or carbides. In general, it is possible to use the metal, a halide or oxyhalide, nitrates or solutions of nitrates, the oxide itself, or, as in one instance in the literature [42], something as unexpected as silicone oil. This variety allows considerable latitude in finding a starting material of appropriate purity and delivery characteristics. Unfortunately, the effect of various starting materials on particle size has not been specifically studied and data available in the literature contains too many other variables to draw any sound conclusions.

The most intense area of ultrafine oxide synthesis has centered on Al_2O_3 and TiO_2. In the synthesis of Al_2O_3 a very wide range of particle sizes from 2-150 nm has been observed with a typically spherical morphology, as shown in Fig. 3. The reported phase composition has

Table III. Plasma Production of Ultrafine Oxides

Compound	Starting Materials	Plasma Type	References
Al_2O_3	$Al/AlCl_3+O_2$	RF/Arc	33,34
Al_2O_3/Cr_2O_3	Al Halide$+O_2$ $+CrO_2Cl_2$	RF	33,35
SiO_2	$SiCl_4+O_2$	RF	37
SiO_2/Al_2O_3	$Si+Al+O_2$	RF	36
TiO_2,TiO_2/Cr_2O_3	$TiCl_4+O_2$ $+CrO_2Cl_2$	RF	38,39
ZnO,Sb_2O_3,BaO SiO_2,MgO	Oxides	Arc	40
MgO	$Mg(NO_3)_2$(aq)	RF	41
ZrO_2,ZrO_2/Al_2O_3	$Zr(NO_3)_2$(aq) $+Al(NO_3)_3$(aq)	RF	43
ZrO_2/SiO_2	$Zr(NO_3)_2$(aq) +Silicone Oil	RF	42

also shown considerable variety. In the synthesis of pure Al_2O_3, Bolotov et al. [34] report the presence of δ, θ, and θ' phases while Barry et al. [33] found γ- and δ-Al_2O_3. Studies of alumina/chromia composites have show δ-Al_2O_3 as the major phase in one case [33] and θ-Al_2O_3 in the other [35]. Available data are insufficient to determine whether the resulting phases are related to the choice of starting materials, the temperature profile in the plasma, the method and location of injection, the method of quenching, or all of the above.

The stabilization of zirconia by additions of Al_2O_3 or SiO_2 has been developed with some success [42,43]. Ono et al. [42] report that ultrafine ZrO_2 undergoes the tetragonal-to-monoclinic transformation at ~670 K; however, codeposition of SiO_2 in the plasma resulted in stability to 1570 K. Kagawa et al. [43] observed similar stabilization of the tetragonal phase when γ-Al_2O_3 was codeposited in the plasma.

METALS. Ultrafine metal particles have been prepared by vaporization of coarser particles, reduction of chlorides and oxides, and decomposition of carbonyls. Ultrafine iron particles [44] with a statistical median size of 10 nm were prepared by passing coarse iron particles through an argon rf plasma. The observed particle morphology has been interpreted to mean that the particles solidified in the gamma phase and then transformed to the alpha phase before finally cooling. Mitrofanov et al. [45] were unable to analyze the pure ultrafine titanium prepared by the vaporization of coarser titanium powders due to their extreme pyrophoricity. Vanadium particles [45]

were produced by injecting metal powder, containing 25 mol% $V_{16}O_3$, into an argon arc plasma. The pyrophoric vanadium was storable under water-free benzene, but oxidized in methanol. The initial oxide in the injected vanadium was absent in the plasma processed powder, presumably due to carbon reduction by the graphite reaction tube.

Boron powders [46] have been prepared from $BCl_3/H_2/Ar$ mixtures in a BCl_3/Ar rf plasma. This reaction scheme produced up to 0.25 kg/hr of 99% boron at 30 kW. The β-rhombohedral particles ranged in size from 20 to 750 nm. Wilks and Lacroix [47] have shown that ultrafine Fe powders can be synthesized by the hydrogen reduction of $FeCl_3$ with yields of 60 to 80%. Neuenschwander [48] has prepared ultrafine tungsten by reducing tungsten hexachloride in a hydrogen plasma. The particle size range of 10-100 nm were obtained, consistent with a measure specific surface area of 9.5 m^2/g.

Several researchers have examined the production of silicon and titanium powders by plasma reduction of a chloride precursor with hydrogen. Coudert et al. [49] have reduced $SiCl_4$ within a hydrogen arc plasma. They obtained 50% efficiency with a 29-kW hydrogen arc and with a $H_2/SiCl_4$ ratio of four. Akashi et al. [50] have described their work on the plasma reduction of $TiCl_4$ with H_2 in an argon plasma jet. Fine titanium crystalline needles were collected on a hot tungsten plate to rapidly quench the titanium vapor. Similarly, a very pyrophoric powder was collected on a water-cooled copper surface. The titanium needles readily reacted with the nitrogen in dry air to form golden-yellow crystals of titanium nitride. Oxygen impurities were found to be undetectable by electron probe microanalysis after a long storage time under dry air.

Plasma reduction of niobium and molybdenum oxides [51] has been carried out with methane/carbon black and hydrogen, respectively, in an argon arc plasma. Processing of an Nb_2O_5/C mixture in the arc column did yield only 65.5% reduction, although the final product can be reduced to pure metal under vacuum reduction. Hydrogen reduction of MoO_2 did reach 81% efficiency with 2.3 times the stoichiometric amount of hydrogen. Both powders oxidized strongly in air. The authors believed that a greater quench rate could substantially improve the degree of reduction.

A pyrophoric iron powder [52] has been prepared from the decomposition of the pentacarbonyl in an argon rf plasma. The resulting powder had a specific surface area of 1.53 m^2/g and an equivalent mean diameter of 0.5 μm. The carbon content was generally less than 1 wt%, reducible to lower levels with the addition of hydrogen to the plasma. Plasma pyrolysis of $Mo(CO)_6$ has been studied for the synthesis of ultrafine Mo powders [53] in a nitrogen rf plasma. The specific surface area of the powders ranged from 16 to 26 m^2/g, which corresponds to a mean particle diameter of 22-36 nm. The amounts of carbon and oxygen in the powders were less 0.2 and 1 wt%, respectively, without the addition of hydrogen. Ultrafine nickel powders [5]

from nickel tetracarbonyl also contain significant amounts of carbon, ranging from 0.37 to 2.2 wt%, and oxygen, typically less than 0.08 wt%. These levels can be easily reduced to less than 0.05 wt% total by hydrogen reduction during sintering. Unfortunately, the ultrafine nickel was highly agglomerated with extensive necking between particles when the carbonyl was injected at rates between 1 and 4 g/min.

ALLOYS. Plasma synthesis of ultrafine alloys has been attempted for a limited number of binary compounds of V, Nb, Cr, and Mo with Si [13,54,55], Nb with Ge [13], Nb with Al [55], and Fe with Al [56]. The ultrafine alloys were typically prepared by injecting a powder mixture of both reactants or a prealloyed powder. The metal alloying is accomplished by vaporizing the reactant mixture and condensing a metal alloy by the rapid quenching in the plasma jet or tail flame. The goal is to quench the metal vapors at a sufficiently rapid rate to condense both metal species simultaneously and promote the alloy formation. A slow quench rate will result in the segregation of the metal vapors into distinct metal phases, as the metal with the higher boiling point will condense first. Therefore, the quenching of the metal vapor is a critical factor in the synthesis of alloys and intermetallic compounds.

Plasma synthesis of a Fe-Al (2.2 wt%) solid solution [56] has been achieved between two elements with similar vapor pressures. The A15 compounds V_3Si and Cr_3Si [13,54] have also been prepared by plasma processing of elements with similar vapor pressures (V:Si and Cr:Si vapor pressure ratios of approximately 1:10 and 10:1 at 2500 K). Yoshida et al. [54] reported that pure V-Si alloys with controlled compositions were prepared in an argon plasma with quenching on a cold surface. Nb-Si, Nb-Ge, and Nb-Al alloys [13,54,55] have proven to be quite difficult to prepare by plasma processing. Typically, multiphase powders produced by plasma synthesis have contained Nb_5Si_3, $NbSi_2$, Nb_3Si, Nb_5Ge_3, $NbGe_2$, and $NbAl_3$ compounds, but the composition of the powders could not be controlled by the composition of the reactant mixture. The ultrafine alloy Nb_3Ge [13] has been prepared by injecting prealloyed powders of this composition, but at a rather low yield of 30 wt%. Interestingly, Ronsheim et al. [13] have successfully produced a Mo_3Si alloy from a 3:1 Mo+Si mixture with a 90 wt% yield in a H_2/Ar arc plasma. Because the alloy could not be produced in an argon plasma, addition of hydrogen may simply increase the heat transfer between the particles and the gas.

The results of these studies show that alloy formation between elements with vapor pressures dissimilar by several orders of magnitude will require faster quenching rates than those previously used. The vapor species must be forced to nucleate simultaneously, if control over the alloy composition is to be achieved.

METAL-CERAMIC COMPOSITES. Plasma synthesis has great potential, although largely unexplored, in the preparation of composite powders

directly in the plasma reactor. Such composite powders could have wide application in the formation of dispersion strengthened metals containing several volume per cent of a ceramic dispersoid. Plasma processing of the composite has three distinct advantages over the mixing of conventional powders and separately prepared plasma powders. The ceramic and metallic particles can be prepared in a submicron size range without extensive and contaminating milling. Also, because of the agglomerates typically present in ultrafine powders, plasma processing from the gas phase can yield a more uniform mixture than that obtainable by milling. Finally, surface metallization of the ceramic particles is a distinct possibility during plasma synthesis by the deposition of a thin metal coating on each particle. This appears to be quite likely as the ceramic particles should typically nucleate before the metal particles do.

Russian researchers [18] have studied the production of ultrafine TiN-Mo composites from $TiCl_4$ and $Mo(CO)_6$ in a nitrogen rf plasma. The composite contains significant amounts of oxygen and carbon from the carbonyl, plus titanium oxycarbonitride. At Los Alamos [5], work has started on the synthesis of Al_2O_3-Ni composites by the injection of Al+Ni powder mixtures into a N_2/Ar rf plasma. Oxygen is added as an O_2/Ar carrier gas for the starting powder. A hydrogen quench gas is necessary to prevent oxidation of the nickel particles. The composites in both studies contain ultrafine particles (<1000 nm) and show excellent mixing between ceramic and metallic particles. However, no evidence has been found in either study to indicate that surface metallization had occurred. Certainly, more research is needed to prove or disprove the possibility of surface metallization.

SUMMARY

We have reviewed plasma chemical synthesis of ultrafine powders for ceramics (nitrides, carbides, and oxides) and metals (metals, alloys, and ceramic/metal composites). The limited examples of plasma processing cited in this review should give some indication of the great potential for this technique in preparing unusual and specialized powders. Although plasma synthesis of ultrafine powders has enjoyed a favorable beginning, considerable research is still needed to develop and refine this synthesis method in such areas as particle size control and particle agglomeration. Also, characterization and dispersive processing of the plasma powders are vitally important for the successful applications of the ultrafine powders, but yet are largely undeveloped for particles of such a fine size.

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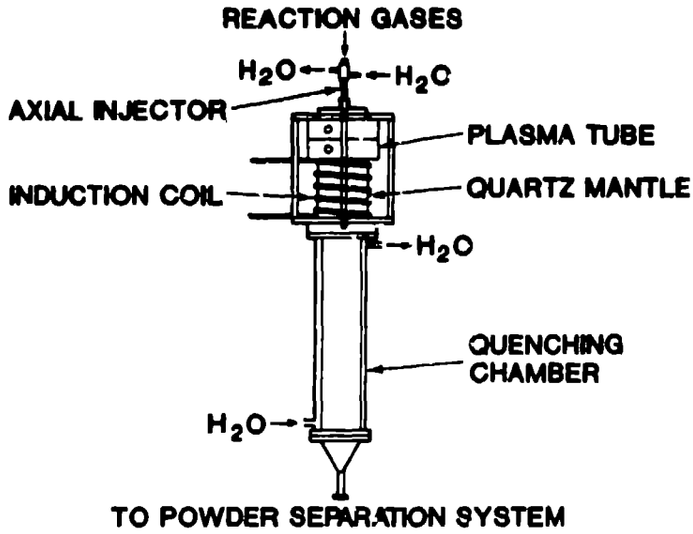

Fig. 1. Schematic of Los Alamos Reactor [5].

Fig. 2. TEM of Los Alamos SiC.

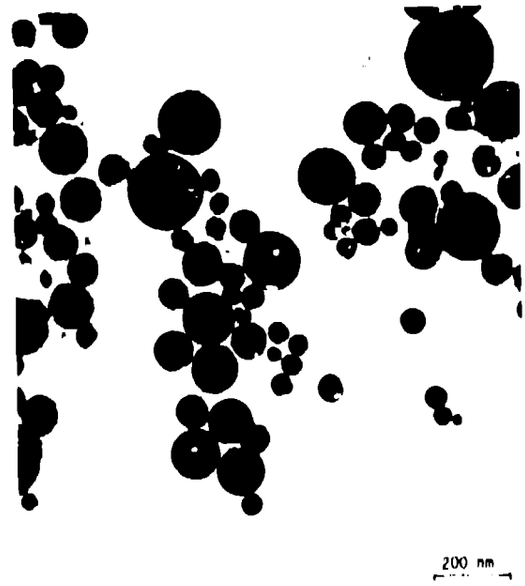

Fig. 3. TEM of alumina prepared from $Al+O_2$.